1906

The Microscope as an Aid in the Heat Treatment of Steel

Augustine Edward Greene

The Microscope as an aid
in the Heat Treatment of Steel

A. E. GREENE 06.

THE MICROSCOPE AS AN AID IN THE HEAT TREATMENT OF STEEL.

Augustine Edward Greene.

I N T R O D U C T I O N .

The gradual but certain displacement of wrought iron for manufacturing purposes by steel of various kinds has compelled manufacturers to more closely study the methods of hardening, tempering, and annealing. This is due to the greater demands of the consumer for strength and durability, and uniformity in texture and composition of the material as a finished product, whether it be steel rails, structural shapes, shafting, car wheels or gun parts.

The old methods of steel treatment are slow and very uncertain in results and frequently valuable amounts of material, purchased according to the most rigid specifications as to the composition in the raw state, were consigned to the scrap heap, owing to the ignorance of the workmen, the lack of uniformity in the material itself, or difficulty in working it into proper shape after being subjected to the crude methods of treatment.

It was believed formerly that if the chemical composition of a metal were known it could be stated whether the metal were of good or bad quality. But it has since been proven that this belief is absurd. "You might", as one eminent metallurgist has said, "just as well say that if a physician knows the amount of carbon, nitrogen, oxygen, hydrogen, etc., in a certain man's body, the state of his general health is known." But we frequently find cases of failure in a material which is as near ideal as possible to make it, and, were it not for the science of Metallography the cause of failure would be very difficult indeed to determine.

C H A P T E R I.

A R T I C L E I.

The science of Metallography was first discovered by H. C. Sorby in 1864, and has since been brought into practical use by Howe, Le Chatelier, Campbell, Sauver and others.

Before we take up the subject of Metallography proper, let us dwell for a moment on the changes which steel undergoes when heated and cooled, but first we will take up the changes in a sodium chloride solution to enable us to better understand the changes of temperature in steel.

If we take a 5% solution of sodium chloride we find its freezing point is below that of pure water, and, if more salt be added, the freezing point is reduced still more and this goes on until the solution contains 23 1/2% of sodium chloride, when further additions of salt raise the freezing point.

This experiment reveals something more interesting. If the solution contains say 5% of salt, pure ice separates out as the solution freezes and, in consequence, the solution which remains contains more than 5% NaCl dissolved in it. The freezing point of the mother liquid is therefore lower than the original solution. This separation of pure ice goes on along the curve AP, Fig. I, until the solution contains 23 1/2% of NaCl. After that, the residual mixture freezes "en masse" at -22°.

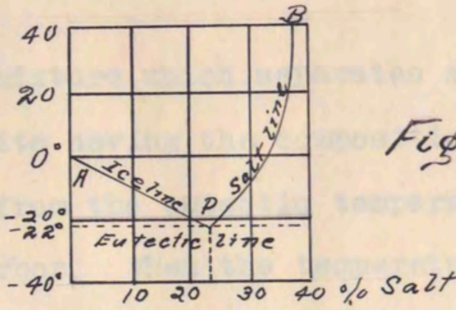
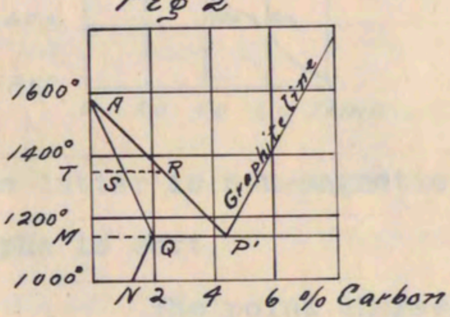


Fig. I

If the solution contains more than 23 1/2% of NaCl then pure salt separates from the solution and the separation of salt and the lowering of the freezing point of the solution goes on along the curve BP (Fig. I) until the solution contains 23 1/2% of salt and the whole residue then solidifies at -22° C. No other mixture of salt and water freezes at a lower temperature than this. Hence this mixture is called a eutectic mixture.

A similar phenomenon occurs when a molten solution of iron and carbon is cooled. If molten iron, containing less than 4 1/2% of carbon, be cooled a solid solution of carbon in iron begins to separate along the line AP' (Fig. 2). This solid solution of carbon in iron is called

Fig 2

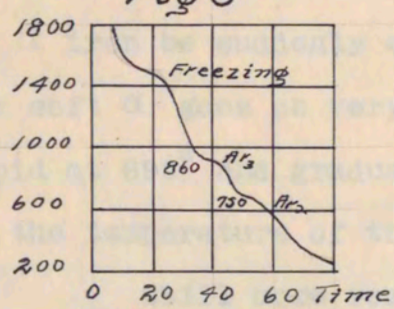


martensite. The solid solution of carbon in iron which separates is not a definite chemical compound. The iron will not retain in solid solution more than 2% of carbon, whereas the molten mass may have as much as 4 1/2% of carbon. We have a new curve AQ (Fig. 2). The abscissa at any point on the curve AQ represents the compositions of the solid, which separates when the solution has the composition represented by the abscissae of the curve AP' corresponding with the same ordinate.

The eutectic mixture which separates at P' consists of graphite associated with martensite having the composition represented by MQ. As the iron cools down from the eutectic temperature OM, the solid martensite rejects more carbon. When the temperature falls to 1000°C. the solid solution of martensite contains 1.8% carbon. The carbon which separates out from the solid metal at temperature below the point of solidification is usually in the form of a very fine powder and is called <u>temper carbon</u>, <u>temper graphite</u>, or annealing carbon; Graphite being an allotropic modification of carbon.— By allotropic we mean the state of a substance when it exists in two or more forms which differ in their properties.—

The cooling curve of iron from the molten condition is shown in Fig. (3) and this, according to Osmond, points out the existence of three allotropic modifications of solid iron:

Fig 3



Alpha Iron - Below Ar_2 or below 750°.

Beta Iron - between 860° - 750°.

and Gamma Iron - above 860°.

The latter is non-magnetic. Gamma iron is very hard but not stable. Alpha is soft.

The point whereeach change takes place is called the critical point and each point is found to be associated with a change in the mechanical properties, the microscopic appearance, the electrical conductivity, the magnetic properties and the specific gravity of the metal.

The critical points are very much affected by the presence of foreign substances. The influence of carbon is shown roughly in Fig. 4.

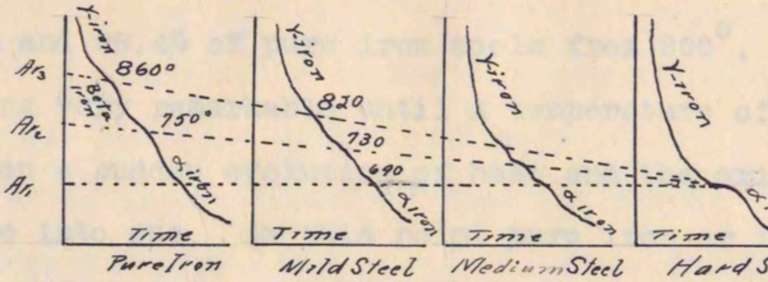


Fig 4

The three critical points Ar_1, Ar_2 and Ar_3 gradually converge into one critical point at about 690° as the % of carbon increases.

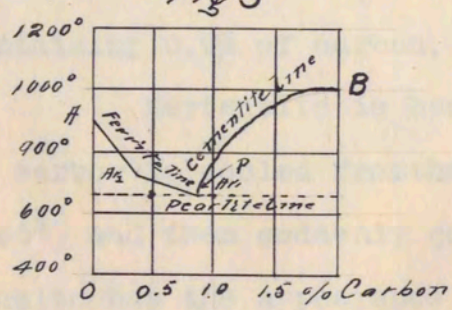
The effect of adding carbon is to increase the stability of hard (γ) iron. Pure γ iron is very unstable at ordinary temperatures and it passes rapidly back to normal α iron at ordinary temperatures. But if a little carbon be present the rate of transformation is reduced and if γ or α iron be suddenly cooled the transformation of the hard γ iron to the soft α goes on very slowly. The rate of transformation is most rapid at 690° and gradually slows down to zero as the metal cools down to the temperature of the surrounding air.

Still more remarkable effects are produced by alloying the metal with manganese, nickel, chromium and tungsten. These elements may lower the Ar_1 point from 690° down to atmospheric temperatures. In the so-called "high speed steels" these alloys are added and have the effect of maintaining a cutting edge at a much higher temperature than ordinary carbon steel.

ARTICLE II.

Let us consider what takes place when an iron bar containing 0.6% carbon and 99.4% of pure iron cools from 900°. The cooling curve shows nothing very remarkable until a temperature of 720° is attained. There is then a sudden evolution of heat and the critical points Ar_3 and Ar_2 coalesce into one. At this point pure iron or ferrite, as Howe calls it, separates from the solid solution. The separation of ferrite goes along the curve AP (Fig. 5) until the temperature reaches about 660° when another rec~~o~~alescence occurs as the system cools down to the normal temperature of the atmosphere. Other alloys of different amounts of carbon furnish a set of curves quite analogous to the freezing curves of salt water, but with iron these changes take place in the solid cooling metal.

Fig 5



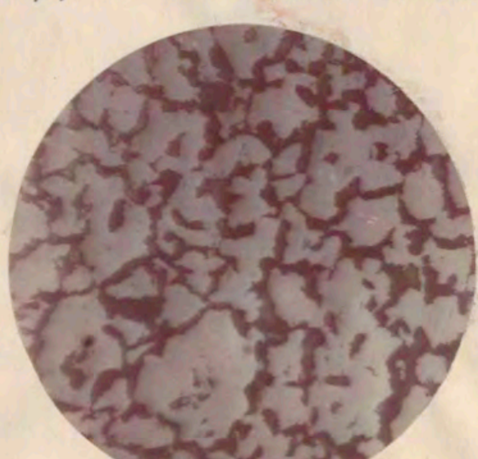
If the alloy contains less than 0.89% of carbon there is a separation, or, better segregation of ferrite; if the alloy contains more than 0.89% of carbon there is a separation, not of carbon, but of a chemical compound of carbon with iron called <u>cementite</u>. Cementite contains 6.9% of carbon. The separation of cementite occurs along the curve BP (Fig. 5). We are therefore dealing with a mixture of ferrite and cementite. The eutectoid alloy contains 13% of cementite and 87% of ferrite. The eutectic mixture of cementite and ferrite is called pearlite owing to the fact that it generally shows the tints of mother-of-pearl under the microscope.

A R T I C L E III.

We have already met with martensite as the solid solution of carbon in iron which separates during the solidification of molten iron. It may contain as much as 2% of carbon above 1130; but, as, as the solution cools, cementite gradually separates out and the remaining solid solution of carbon becomes poorer and poorer in carbon. The separation of cementite continues until the solid solution has 0.9% of carbon. This is the eutectoid mixture which segregates into pearlite. Some reserve the term martensite for the unsegregated eutectic mixture containing 0.9% of carbon.

Martensite is best formed when a steel containing 0.2 to 0.8% of carbon is cooled fromthe Ar_3 critical point (830°) slowly to Ar_2 point (730°) and then suddenly quenched in a freezing mixture at - 20°. Martensite has the appearance of interlacing needles.

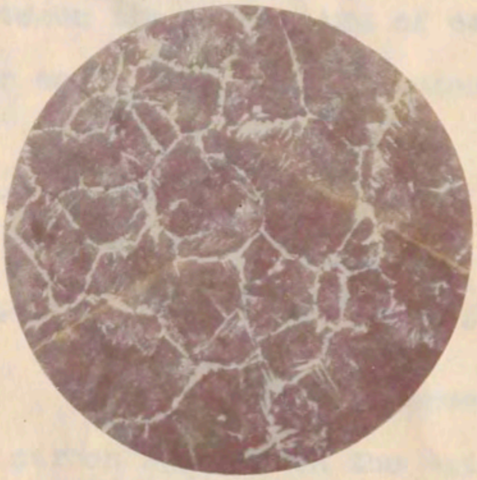
When steel has just the eutectic proportion of carbon, namely 0.9%, the martensite is called Hardenite.



Forged Steel showing Martensite and Troostite Carbon = 0.50% Magnified 100 dias. Quenched during Critical Temp.

If the temperature at which pearlite segregates be hastened by quenching the hot metal in lead, the pearlite loses its well defined lamellar appearance and we get what is called sorbitic pearlite or sorbite. Sorbite is unsegregated pearlite. Some say that what we have called granular pearlite is really sorbite. Whatever sorbite may be it is not homogeneous.

There is another transitional form between martensite and pearlite. It is produced when a metal containing about 50% of carbon is cooled down to the Ar_1 point (690^{o}) and quenched in water at atmospheric temperature. This constituent is called Troostite.



Sorbitic Steel
Carbon = 0.55 %
Magnified 100 dia's.

There are therefore two transitional forms between the martensite and pearlite stages of cooling steel.

Martensite → Troostite → Sorbite → Pearlite.

Many metallurgists maintain that troostite and sorbite only refer to particular patterns which the constituents of the cooling alloy assume when the metal is quenched under special conditions.

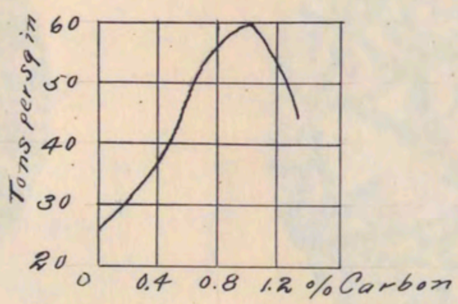
C H A P T E R II.

A R T I C L E IV.

The mechanical properties of iron-carbon alloys are closely connected with the relative amounts of the two elements. The relation between the percentage of carbon in an alloy and the tenacity in tons per square inch is indicated in the following table:-

Percent. of carbon	*0.05*	*0.1*	*0.2*	*0.4*	*0.6*	*0.8*	*1.0*	*1.3*
Tenacity	*25.00*	*26.00*	*31.0*	*36.0*	*43.0*	*58.0*	*60.0*	*44.0*

The gradual increase in the tenacity of the metal as the amount of carbon approaches the eutect*oid* proportions is brought out very closely. The results are shown graphically in Fig. 6.



Cementite is a very brittle substance, harder than glass, while ferrite is soft and as ductile as copper. The relative proportions and the distribution of these two constituents in any alloy must affect its mechanical properties. In hypoeutectic steels the presence of an excess of ferrite renders the metal ductile and tenacious. On the other hand, in hypereutectic steels the presence of an excess of cementite diminishes the ductility of the metal.

When the metal has 2 or more % of carbon the resulting alloys are called cast irons. White cast iron for example has so much cementite that it cannot be filed or drilled with ordinary tools. In white cast iron the carbon is principally in the form of cementite. By heating white cast iron to a suitable temperature the cementite is decomposed into free graphite carbon. The result is gray cast iron in which the carbon is not combined with the iron but is present in the form of graphite carbon. The effect of free graphite on the properties of the alloy is mechanical only. It destroys the continuity of the metal and so renders it liable to fracture under the influence of mechanical stresses. Gray cast iron is soft enough to be worked in mechanical operations.

White cast Iron

#1 Grey Cast Iron

A R T I C L E V.

The structure and properties of steel and cast iron may be greatly modified by the rate at which the metal is cooled from a high temperature. Two pieces of steel having exactly the same *composition* chemically may have entirely different physical properties. The hardness, tenacity and other properties of the metal depend upon the relative proportions of the allotropic modifications of iron and of martensite, ferrite and cementite present in the metal; and these proportions in turn depend upon the rate of cooling. There is a particular temperature at which the speed of the transformation of hard martensite into soft pearlite goes on most rapidly; and if the metal be cooled down past this temperature before the hard martensite has time to pass into soft pearlite then the passive resistance to further change becomes so great that any further change is arrested and the properties of the hard martensite will predominate. On the other hand, if the metal be cooled down past this critical temperature very slowly, the martensite will pass into pearlite, and the properties of the metal will be altered accordingly. The physical properties of the metal thus depend upon:

(1) Its chemical composition

(2) The heat treatment to which it has been subjected.

The sole object of hardening, tempering and annealing steel is to make the metal pass through the various critical temperatures with the proper velocity; in other words, the different transformations which take place at the critical points are arrested when the constituents are distributed in the proportions necessary to confer upon the metal the required degree of hardness. When it is remembered how dependent the rate of cooling of a mass of metal is upon external conditions - specific heat, thermal conductivity of the quenching fluid - it is easy to see how so many empirical directions for the tempering of steel for special purposes have crept into metallurgical practice.

It may also be noticed that for the uniform tempering of a mass of steel it is necessary for the whole mass to undergo the same variation in temperature! This is hardly possible in practice because the cooling must go on through the outer surface of the metal, but the rate of cooling increases as the square of the surface, while the total quantity of heat to be removed increases as the cube of the mass of the metal. Hence the difficulty of hardening successfully large masses.

ARTICLE VI.

The fracture of a metal, or the broken surface which the metal presents, may be fibrous or crystalline. Experts can deduce a considerable information from the appearance presented by the fractured cross-section of a metal. Wrought iron has a fibrous fracture, while cast iron and steel have a peculiar crystalline fracture for every variety of the metal.

The fracture, when performed under definite conditions, furnishes a true indication of the coarseness of crystallization. The degree of coarseness of the fracture or the average size of the crystalline grains when a suitably prepared specimen is examined under the microscope, is called the grain size of the specimen. As a general rule the smaller the grain size the better the steel. An experienced man can generally give a very accurate guess of the temperature to which a metal has been heated from the fracture or from the microstructure of the metal since it is a well established fact that the higher the temperature above the critical point to which the metal has been heated the larger the grain size. The steel will have the finest grain if heated to the critical point and then cooled from this point.

If hardened or unhardened steel be heated to the Ac_1 critical point all previous crystalline structure, however coarse and distorted is obliterated and replaced by the finest possible structure the metal can assume. This rearrangement of the size of the grains is called heat refining. The breaking up of the old structure is due to the change of cementite into martensite and to the diffusion of carbon after these changes. The transformation of cementite into martentite is sudden: while the passage from martensite takes place gradually and is accompanied by the evolution of heat.

This law only applies to hard steels. With hypoeutectic steels the old coarseness is not quite obliterated when the Ac_3 point (on the cooling curve) is reached and as the old structure being destroyed between the Ac_1 and Ac_3 point a new growth sets in. Consequently hypoeutectic steels cannot be refined as completely as eutectic or hypereutectic steels because the old structure cannot be effaced without permitting at the same time a considerable growth of the new. J. E. Stead has shown that with very soft steels the ferrite grain grows larger as the temperature rises above 500° and instead of being refined the grains grow until the temperature of 900° is reached when the former granular structure is broken up and the steel is refined. This shows how necessary it is to avoid heating soft steel for any length of time at 500°.

If steel be heated for some time to a temperature just above 1000°, the metal, even mild steel, becomes very brittle and acquires a coarse crystalline structure. The size of the crystal may be considerably reduced by quickly cooling the steel from the temperature of overheating, although brittleness may still exist. Steel so affected is said to be overheated. The disease can be cured by heating the steel for a few days near the Ac_3 critical point (900°).

C H A P T E R III.

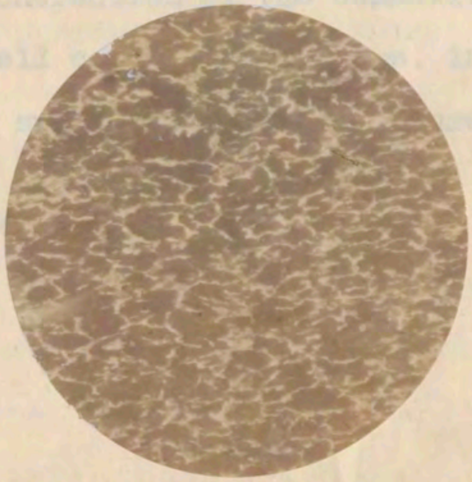
A R T I C L E VII.

The important structural changes noticed by the aid of the microscope during the working of steel are as follows.

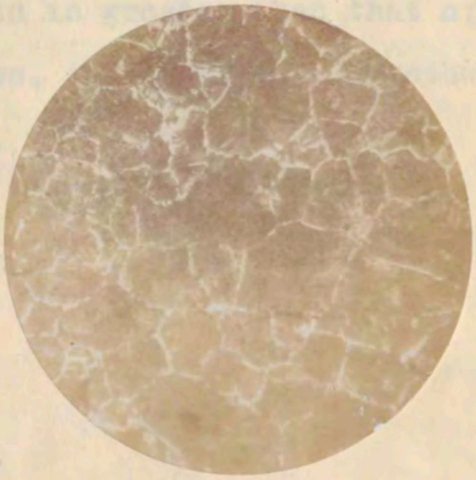
I. If the work is sufficiently vigorous to affect all parts of the mass no crystallization takes place while the metal is being worked.

II. Hot working of the steel has no direct action upon the structure of the steel but as it retards crystallization until a lower temperature is reached it may influence structure in this way.

III. Cold work distorts the grain or flattens and elongates the srystals in the direction of rolling. The lower the temperature the more pronounced the effects of cold working. The structural changes of cold working can be obliterated by heat treatment up to or past the Ar_1 critical point.



Cold Worked Steel
Shows distortion of grains in direction of drawing
C = 0.50 %
Mag. 100 Dia's.



Cold Worked Steel
after annealing
Heated to 1000°C and cooled in furnace C = .50 %
Mag. 100 dia's.

A R T I C L E VIII.

Occasionally we find cases of steel failing from no apparent reason whatever when subjected to a blow which under ordinary conditions is far below that which would cause rupture. This may not be due to any alteration in the crystalline form of the metal. The normal state of steel is crystalline. In many cases the source of weakness is the joints between the crystalline grains. When such a metal is fractured the line of fracture follows the junction of the grains. One per cent of iron sulphide will entirely destroy the ductility of iron, reducing its ultimate stress from 20 to 2 tons per square inch.

The net work of cementite which envelopes the crystal grains of steel containing more than one per cent of carbon are the principal lines of weakness. The metal, when fractured, generally breaks through the center of this brittle envelope. The coefficient of contraction of the cementite cell walls is greater than that of the cell contents. The mass, in consequence, is feebly held together and a sudden blow will fracture the metal.

A R T I C L E IX.

From what has preceded it is obvious that the work of the microscope consists essentially in the definition of structural shapes, crystalline or granular forms. Since the microstructure has been shown to depend upon the chemical composition and the heat and mechanical treatment of the metal, and, furthermore, the physical properties also being dependent upon and modified by the same means it has led to the hope that the microscope might be found a practical and reliable aid in judging those physical properties which are made use of in the constructive arts.

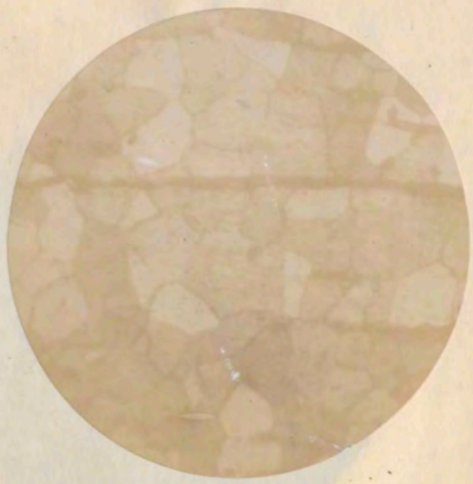
The microscope itself carries with its appearance no suggestion of the tensile properties nor the limit of endurance to repeated stresses which accompany a given piece of steel. It is through comparisons of structure with the results of the testing machine that knowledge of the forms seen acquires value. That is to say, the microscope is an indirect agent in judging the physical properties of the metal and its position in the test, and the acceptance of materials for engineering purposes must be established by a series of concurrent observations with a direct method.

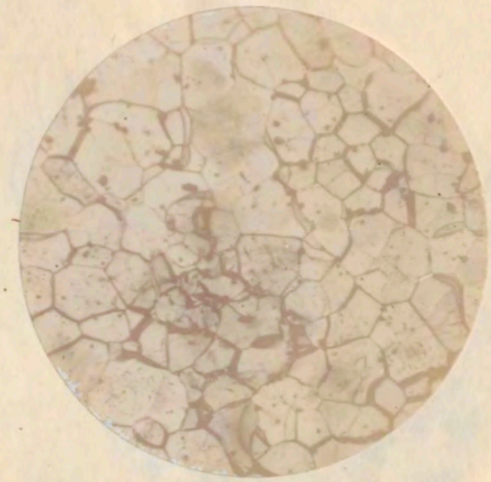
In judging the structure of steel it is more satisfactory to make an examination in the presence of the material at the microscope than through the medium of photography. Some structures are easily reproduced and photographs satisfactorily represent the material, while in other cases photographic methods are distinctly disappointing in results, since only a small part of the surface of the material can be exhibited and the selection of the particular spot to best represent the structure is not infrequently perplexing.

The microscope should not be relied upon wholly but it should be supplemented by chemical and physical tests. However, in hardening and annealing steel the microscope alone can be used to determine what the treatment has been since in the machining of small machine parts the question of strength is not often considered since the dimensions are usually very ample. It is of great importance, however, that the material be in such condition as to permit rapid work with the least wear upon the tools used and also to determine whether the steel in the tool is faulty or whether it is the parts being machined.

Then we may conclude by saying that the microscope is not to be trusted too explicitely in the examination and determination of the specifications for structural steel but should always be supplemented by chemical and physical tests, but in the case of steel which is used for machine parts the microscope is invaluable.

Below are given several micrographs which showed many of the characteristics of steel and iron and the effect of heat treatment.

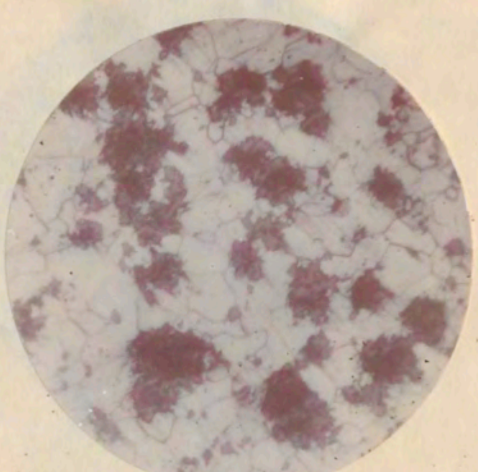




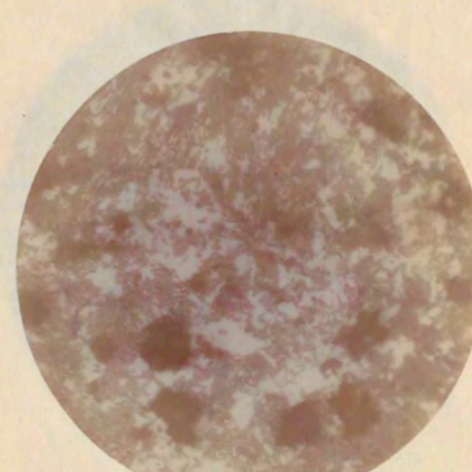
Comercial Wrought Iron

Mag. 100 dia's.
Longitudinal Section.
Showing Slag lines

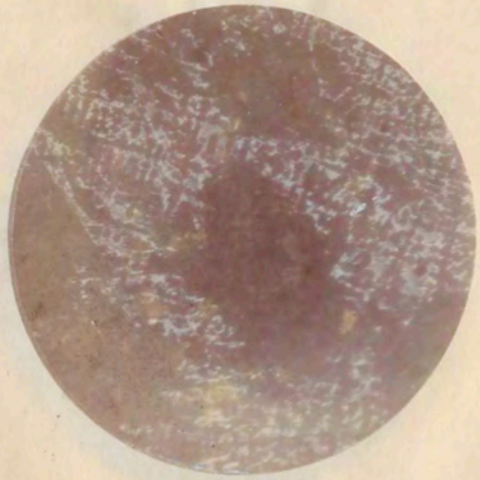
Cross Section
Mag. 100 dia's.



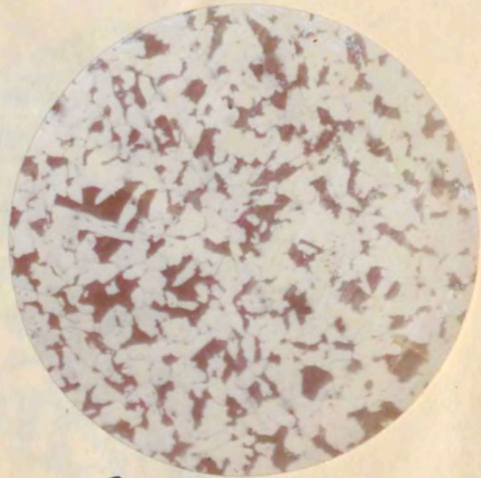
Malleable Cast Iron
Carbon change complete
Mag 100 dia's.



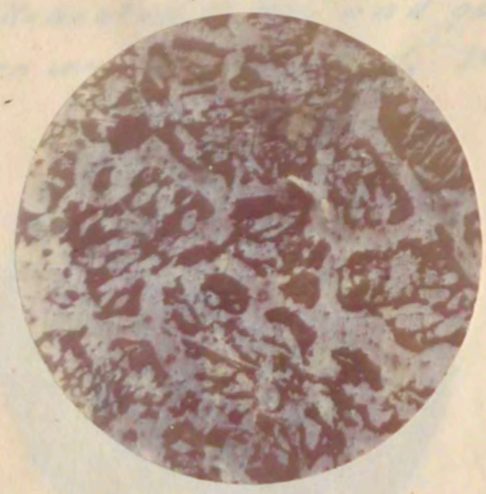
Malleable Cast Iron
Carbon change complete
Mag 100 dia's.



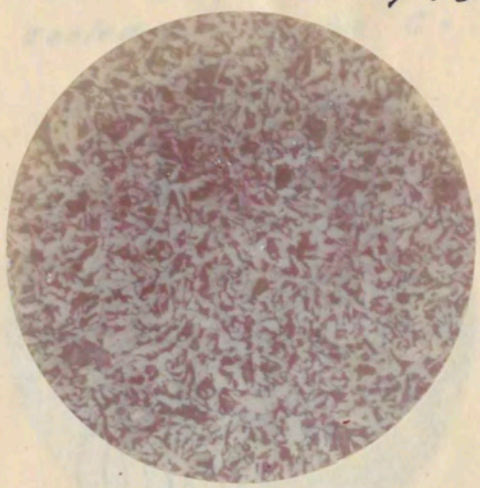
Mottled Cast Iron
Mag. 40 dia's.



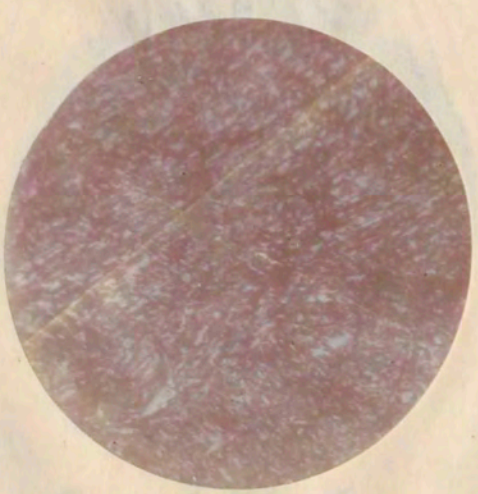
C = .30 %
Forged Steel reheated
to 1000° and cooled with furnace
Mag. 100 dia's.



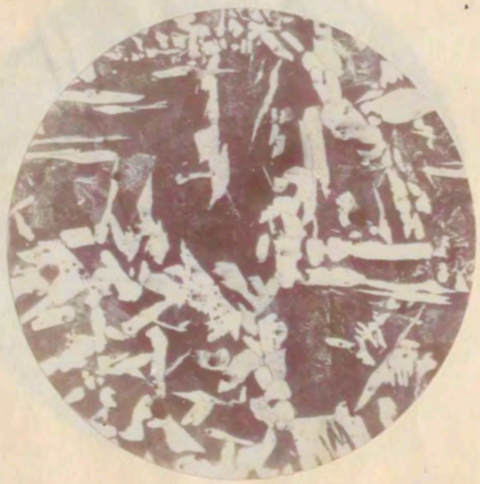
Steel as Cast
C = .30 %
Mag. 100 dia's.



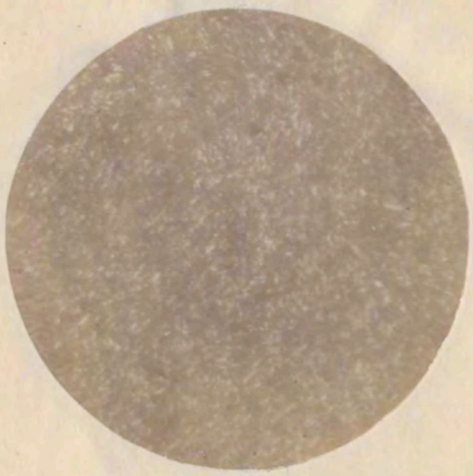
Cast Steel Annealed
Heated at 800°C and cooled
in air. Heated for two hrs.
C = .30 % Mag. 100 dia's.



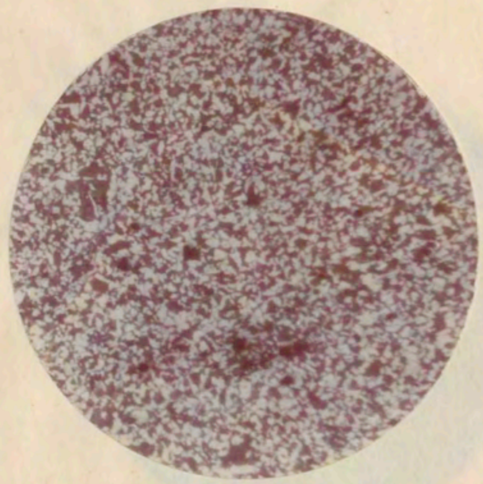
Forged Steel
Reheated to 800° and quenched
in water C = .50 % Mag 1000 dia's.



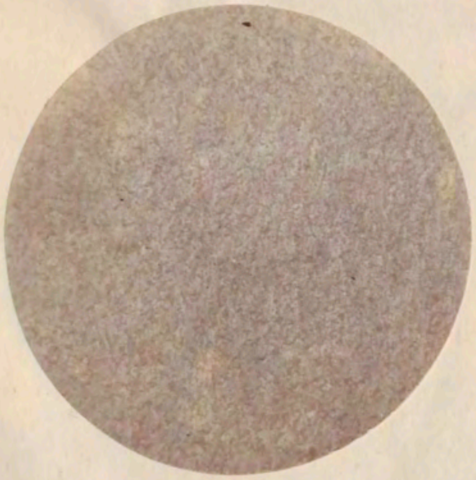
Forged Steel Mag. 100 dia's.
Reheated to 1100° and
cooled in furnace C = .50 %



Forged Steel
Reheated to 800° and quenched
" " 600 " "
C = 50 % Mag. 200 dia's.

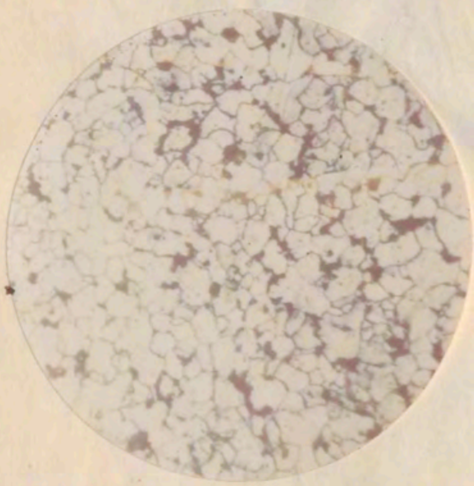


Forged Steel
Reheated to 800° and cooled
in furnace C = .54 % Mag 100 dia's.

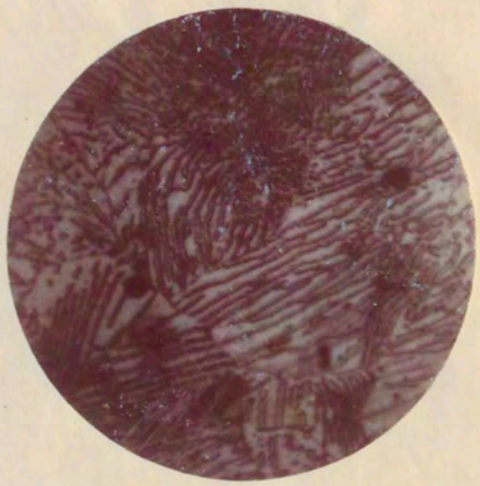


Forged Steel
Hot worked - Not treated
C = .3 % Mag. 100 dia's.

Crucible Steel
Reheated to 1100 and
cooled with furnace C = 1.25 %
Mag 500 dia's.



Forged Steel
Reheated to 1000° and
cooled with furnace
C = .10 % Mag. 100 dia's.



Crucible Steel
Reheated to 1000° C
and cooled in furnace
C = .96 % Mag. 1000 dia's.